



## Research Paper

# Ascorbic acid- and sodium ascorbate-loaded oxidized potato starch films: Comparative evaluation of physicochemical and antioxidant properties



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## ABSTRACT

The purpose of this study was to compare the effects of increasing concentrations of ascorbate ions (AIs, 0–100 mM) in the form of ascorbic acid (AA) and sodium ascorbate (SA) on the properties of edible oxidized potato starch films. The browning reactions were faster in the SA-added films than in those of AA-added. In turn, AA recrystallized faster than its sodium salt. The highest concentration of SA increased the water vapor permeability of the films. The mechanical strength and stiffness of the films gradually decreased with the increase of AI content. Fourier-transform infrared spectroscopy spectra suggested that addition of SA provoked a more intensive structural changes in the films than AA. X-ray diffraction showed that 25 and 50 mM AI-added films exhibited higher crystallinity than the control. The films with AA and SA did not differ in terms of dissolving behavior, ability to release AI, and consequently, antioxidant activity.

## 1. Introduction

Exposure to oxygen is one of the main factors that can cause foods to spoil faster. Popular strategies used to limit oxidative degradation are direct addition of antioxidants or reduced oxygen packaging. A novel alternative to these methods is active antioxidant packaging, whose main advantage is that it can provide sustained release of antioxidants during storage (Gómez-Estaca, López-de-Dicastillo, Hernández-Muñoz, Catalá, & Gavara, 2014).

Vitamin C is a powerful antioxidant found mainly in fresh fruits and vegetables. On an industrial scale AA is produced from glucose (Pappenberger & Hohmann, 2014). Many, if not all of the biologic properties of L-ascorbic acid (AA) are based on hydrogen donation abilities (FAO, 2011). With the electron transfer, AA very rapidly scavenges the free radicals. Moreover, AA acts as an oxygen scavenger and chelator of prooxidative metal ions (Brewer, 2011). Cheap mass-production and broad spectrum of antioxidant properties render AA the preferred component in the active packaging systems (Kowalczyk,

2016).

A variety of materials can be used as a matrix holding AA. In response to the urgent need for eco-friendly packaging materials, AA is willingly incorporated into packaging based on biodegradable polymers. Depending on the material origin, treatment and fabrication route, bio-based packaging can be decomposed in the environment, as well as in the human tract, which opens up new opportunities for the food industry. Current uses of “biodegr(edible)” packaging include casing, capsules, coatings, wrappers, water-soluble bags for pre-portioned foods, layers separating various components in complex food, controlled-release dosage forms and cups. AA-added edible coatings primarily limit quality deterioration occurring during storage of fat-rich food, e.g. nuts (Haq, Azam, & Hasnain, 2015; Min & Krochta, 2007), but they can also inhibit enzymatic browning of fresh-cut fruits (Saba & Sogvar, 2016). A recent study (Kowalczyk, 2016) has shown that among the different biopolymers, the films based on oxidized potato starch (OPS) provided the best stability of AA, indicating that the low pH and low water content in the matrix are important factors to

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prevent degradation of vitamin C. AA is a cyclic lactone (ester) containing an endiol group ( $-\text{C}(\text{OH})=\text{C}(\text{OH})-$ ). The compound is acidic, as a consequence of electron delocalization in the ring system, making the OH group-labeled 3-prone to giving off its proton when in contact with neutral or basic solutions (Berg, 2015). AA is sour, thus its incorporation into edible coating could have negative impact on the taste of certain foods (Kowalczyk, Zięba, Skrzypek, & Baraniak, 2017), which however can be overcome by use of ascorbate salts. Since the bitter taste of calcium and potassium ascorbate could be a significant problem for application (Burdock, 1996), the purpose of this study was to obtain the OPS films loaded with sodium ascorbate (SA). The slightly salty/soapy taste of SA should not generally be a problem at current typical usage rates (Burdock, 1996; Madhavi, Deshpande, & Salunkhe, 1995). The properties of SA-loaded films were compared with those of the AA-added.

## 2. Materials and methods

### 2.1. Materials

Oxidized potato starch LU-1404-3 was purchased from WPPZ S.A. Luboń (Poland). 2,2'-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (ABTS), 2,6-dichloroindophenol sodium salt hydrate, ( $\pm$ )-6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid (Trolox), L-ascorbic acid, sodium L-ascorbate, glycerol,  $\text{FeCl}_3$ , potassium ferricyanide (III), trichloroacetic acid, and Tween 40 were purchased from Sigma-Aldrich. All other chemicals were of analytical grade.

### 2.2. Film preparation

Films were obtained from 5% (w/w) OPS solutions containing 1% (w/w) glycerol. OPS was mixed with glycerol and water, then heated in a water bath at 90 °C for 30 min with constant stirring. After cooling to 40 °C, AA and SA were introduced into film forming solution (FFS), so the final concentrations of ascorbate ion (AI) were 25, 50 and 100 mM. The formulation without AA/SA served as the control. The FFSs were cast onto leveled polycarbonate trays (with the area of 4.0 and 144.0  $\text{cm}^2$ , depending on the test) and dried at 50% relative humidity (RH) and 25 °C for 24 h. A constant amount (0.011 g) of total solids was cast onto 1  $\text{cm}^2$  of tray area, in order to maintain film thickness.

### 2.3. Measurement of pH

Flat surface electrode (Elmetron EPX-3, Poland) connected to a pH meter (Elmetron CPC 401, Poland) was used for pH measurement of the FFSs and the casted films after their a slight surface hydration with 20.0  $\mu\text{L}$  of deionized water.

### 2.4. Film thickness and conditioning

Thickness of the films was determined using a manual micrometer (Mitotuyo No. 7327, Japan). Before testing, film specimens were conditioned for 24 h in a test chamber (MLR-350, Sanyo Electric Biomedical Co. Ltd., Japan) at 50% RH and 25 °C.

### 2.5. Fourier-transform infrared (FTIR) spectroscopy

FTIR tests were carried out by using a PerkinElmer infrared spectrometer (PerkinElmer SP 100, USA). Attenuated Total Reflectance (ATR) spectra were obtained in the 4000–600  $\text{cm}^{-1}$  region using 16 scans and 1  $\text{cm}^{-1}$  resolution. All the readings were performed at room temperature (20 °C, 50% RH).

### 2.6. Moisture content (MC) and total soluble matter (TSMc)

Film specimens (4  $\text{cm}^2$ ) were weighed ( $\pm 0.0001$  g) and dried in an oven at 105 °C for 24 h to a constant weight. The weight loss of each sample was determined, and the MC was calculated as the percentage of water removed from system. The TSMc was expressed as the percentage of film dry matter solubilized after 24 h immersion in water. Conditioned film specimens were shaken in 30 mL of distilled water at  $25 \pm 1$  °C for 24 h, then the undissolved film residues were removed from water and dried at 105 °C for 24 h to determine solubilized dry matter. Initial dry matter values needed for TSM calculations were obtained from MC measurements. Analyses were performed in quadruplicate.

### 2.7. Water vapor permeability (WVP)

The WVP ( $\text{g mm m}^{-2} \text{d}^{-1} \text{kPa}^{-1}$ ) was calculated as:

$$\text{WVP} = (\text{WVTR} \times L) / \Delta p \quad (1)$$

where WVTR is the water vapor transmission rate of film ( $\text{g m}^{-2} \text{d}^{-1}$ ), L is the mean of film thickness (mm),  $\Delta p$  is the water vapor pressure difference (kPa) between two sides of the film. The WVTR of the films was measured gravimetrically based on ISO 2528 (1995) method. The permeation cell (poly(methyl methacrylate)) cups had an internal diameter of 7.98 cm (exposed film area = 50  $\text{cm}^2$ ) and an internal depth of 2 cm. Distilled water (30 mL) was added into each test cup and film samples were placed over the circular opening and secured by a screw top. The cups were placed in a test chamber set at 25 °C and 50% RH. The weight loss from the samples with 3 replicates was monitored for 10 h period with weights recorded at 2 h intervals.

### 2.8. Optical parameters

Color values (CIE  $L^*a^*b^*$ ) of the films stored without light in a test chamber (50% RH, 25 °C) were measured with a colorimeter (NH310, 3nh, China) on the white background ( $L^* = 88.4$ ,  $a^* = 2.2$ ,  $b^* = -2.0$ ). The measurements were performed at least three times at random positions on samples. The light transmission (T%) of films was measured at selected wavelengths between 200 and 800 nm. The opacity of the films was calculated by Eq. (2) (Han & Floros, 1997):

$$\text{Opacity} = A_{600} / x \quad (2)$$

where  $A_{600}$  is the absorbance at 600 nm and x is the film thickness (mm). Analyses were performed in triplicate using a spectrophotometer (Lambda 40, Perkin-Elmer, Shelton, CT, USA).

### 2.9. Degree of crystallization of AA and SA

Film samples (12 × 12 cm), both remained on and peeled off the casting surface, were stored in the test chamber (50% RH, 25 °C) for four weeks. The percentage area of AA and SA crystals (white spots) appearing on the film surface was estimated using a histogram tool of image editing software, by counting the number of white pixels exposed in black-and-white photos of the films (Kowalczyk & Baraniak, 2014). Analyses were performed in triplicate.

### 2.10. Microscopy

The scanning electron microscope Carl Zeiss Ultra Plus (Oberkochen, Germany) was used for the inspection of the surface of films. Imaging of samples was performed in high vacuum ( $5 \times 10^{-3}$  Pa) using a secondary electron detector at 20 kV. The surface of films stored for 7 days at 50% RH and 25 °C was observed by LEICA 5500B light microscope connected to a Leica DFC500 camera (Leica Microsystems GmbH, Germany). A polarized light was used to

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